भारतीय मानक

Indian Standard

IS 2600 (Part 6) : 2022

डाई कॉस्टिग के लिए जिंक एवं जिंक आधारित मिश्रणों के रासायनिक विश्लेषण की विधियाँ

भाग 6 एटोमिक अब्जर्पशन स्पेक्ट्रोमेट्री विधि द्वारा मैगनेसियम ज्ञात करना

(पहला पुनरीक्षण)

Methods of Chemical Analysis of Zinc and Zinc Base Alloys for Die Castings

Part 6 Determination of Magnesium by Atomic Absorption Spectrometric Method

(First Revision)

ICS 77.120.60

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Methods of Chemical Analysis of Metals Sectional Committee, MTD 34

FOREWORD

This Indian Standard (Part 6) (First Revision) was adopted by Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Metals Sectional Committee had been approved by Metallurgical Engineering Division Council.

This standard was first published in 1964 and has been revised (in parts) on the basis of implementation of the standard by the manufacturers and the testing.

This standard (Part 6) cover determination of magnesium by atomic absorption spectrometric method in zinc and zinc based alloys for die castings.

This Indian Standard is published in several parts. The other parts in this series are:

Part 1 Determination of copper, iron, nickel, tin and thallium by spectrophotometric;
Part 2 Determination of copper, iron, lead and cadmium by atomic absorption spectrometric method;
Part 3 Determination of indium by atomic absorption spectrometric method;
Part 4 Determination of chromium by spectrometric method;
Part 5 Analysis by inductively coupled plasma optimal emission spectrometry; and
Part 7 Determination of aluminium by titrimetric method.

In reporting the result of a test or analysis made in accordance with this standard, is to be rounded off, it shall be done in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in rounded off value should be the same as that of the specified value in this standard.

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF ZINC AND ZINC BASE ALLOYS FOR DIE CASTINGS

PART 6 DETERMINATION OF MAGNESIUM BY ATOMIC ABSORPTION SPECTROMETRIC METHOD

(First Revision)

1 SCOPE

This standard (Part 6) covers the method for determination of magnesium by atomic absorption spectrophotometric method for the analysis zinc and zinc alloys in the range as specified in IS 209, IS 713 and IS 742.

2 REFERENCE

The following standards contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subjected to revisions, and parties to agreement based on these standards are encouraged to investigate the possibility of applying the most recent edition of the standards indicated below:

IS No.	*Title*
209 : 1992	Zinc ingot — Specification (*fourth revision*)
265 : 2021	Hydrochloric acid (*fifth revision*)
713 : 1981	Zinc based alloy ingot for die casting (*second revision*)
742 : 1981	Zinc based alloy die casting (*second revision*)
1817 : 1961	Methods of sampling non-ferrous metals for chemical analysis

3 SAMPLING

The samples shall be drawn and prepared in accordance with the procedure laid down in IS 1817.

4 QUALITY OF REAGENTS

4.1 Unless otherwise specified, analytical grade reagents and distilled or demineralized water shall be employed in the test.

4.2 Reagents

4.2.1 *Hydrochloric Acid*, rd = 1.16 (*see* IS 265).

4.2.2 *Nitric Acid*, rd = 1.4.

4.2.3 *Hydrochloric Acid/Nitric Acid Mixture* — Mix 180 volumes of hydrochloric acid (*see* **4.2.1**) with 4 volumes of nitric acid (*see* **4.2.2**). This mixture shall be freshly prepared just before use.

4.2.4 *Lanthanum, 5 Percent Solution* — Put 29 .5 g of lanthanum oxide (La_2O_3) in a 400 ml beaker. Add 5 ml of water, and then carefully add 50 ml of hydrochloric acid (*see* **4.2.1**). After dissolution, cool to room temperature. Transfer quantitatively to a 500 ml volumetric flask. Dilute to the mark with water and mix.

4.2.5 *Zinc, 10 g/l Solution* — Dissolve 10 g of zinc (**99.99** percent), free of magnesium (see **5.2.1**), with 60 ml of the acid mixture (*see* **4.2.3**). Evaporate to a syrupy consistency. Take up with water and transfer quantitatively to a 1 000 ml volumetric flask. Dilute to the mark with water and mix

4.2.6 *Aluminium, 1.0 g/l Solution* — To 1.0 g of aluminium (99.99 percent), free of magnesium (*see* **5.2.1**), add 10 ml of water and then dissolve with a minimum of hydrochloric acid (*see* **4.2.1**). Heat it gently to aid dissolution. Cool to the room temperature. Transfer quantitatively to a 100 ml volumetric flask. Dilute up to the mark with water and mix. After verification of the magnesium contents (*see* **5.2.1**), transfer exactly 50 ml to a 500 ml volumetric flask. Dilute to the mark with water and mix.

4.2.7 *Magnesium*, *0.5 g/l Standard Solution* — Into a 250 ml beaker covered with a watch-glass, pour 20 ml of water, then 5 ml of the hydrochloric acid (*see* **4.2.1**). Add 0.5 g of magnesium of purity at least 99.95 percent, weighed to ± 0.001 g. After dissolution of the metal, cool and transfer quantitatively to a 1 000 ml volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 0.05 mg of magnesium.

4.2.8 *Magnesium, Standard Solution, 0.01 g/l* — Transfer exactly 20 ml of the magnesium solution (*see* **4.2.7**) to a 1 000 ml volumetric flask. Add 5 ml of the hydrochloric acid (*see* **4.2.1**). Dilute to the mark with water and mix.

1 ml of this solution contains 0.01 mg of magnesium.

4.2.9 *Magnesium Standard Solution, 0.001 g/l* — Transfer exactly 50 ml of the standard magnesium solution (*see* **4.2.8**) to a 500 ml volumetric flask. Add 5 ml of the hydrochloric acid (*see* **4.2.1**). Dilute to the mark with water and mix.

1 ml of this solution contains 0.001 mg of magnesium.

4.2.10 *Aqua Regia* — Mix 3 volumes of hydrochloric acid (*see* **4.2.1**) with 1 volume of nitric acid (*see* **4.2.2**).

5 PROCEDURE

5.1 Preparation of Sample Solution

Weigh 5 g of the sample to the nearest 0.001 g. Transfer the sample into a 250 ml beaker and cover with a watch glass, and dissolve by carefully adding 40 ml of the acid mixture (*see* **4.2.3**). Evaporate carefully to a syrupy consistency and, after cooling to room temperature, dilute with 40 ml to 50 ml of water. Add 25 ml of the hydrochloric acid (*see* **4.2.1**) and warm gently to dissolve any salts. Transfer to a 250 ml volumetric flask. Dilute to the mark with water and mix. Transfer exactly 10 ml of this solution to a 100 ml volumetric flask. Add 4 ml of the hydrochloric acid (*see* **4.2.1**) and 5 ml of the lanthanum solution (*see* **4.2.4**). Dilute to the mark with water and mix.

5.2 Preparation of Calibration Solution

5.2.1 To verify that the magnesium content of solutions (*see* **4.2.5**) and (*see* **4.2.6**) is low enough, proceed as follows:

a) Introduce, into two 100 ml volumetric flasks, 0 ml and 2 ml respectively of the standard magnesium solution (*see* **4.2.9**) corresponding to 0 ml/l and 0.02 mg/l of magnesium;

b) Dilute to the mark with water and mix; and

c) Compare solutions (*see* **4.2.5**) and (*see* **4.2.6**) with these calibration solutions by spectrophotometric measurement of the atomic absorption as specified in **5.3**. The spectrometric measurement response shall not exceed that of the 0.02 mg/l Solution.

5.2.2 Introduce 5 ml of the hydrochloric acid (*see* **4.2.1**) into each flask of a series of

Eight 100 ml volumetric flasks.

5.2.3 Add 'a' ml of the zinc solution (*see* **4.2.5**) and 'b' ml of the aluminium solution (*see* **4.2.6**) to each flask, according to Table 1.

Table 1 Volumes a and b

(*Clause* 5.2.3)

Sl No.	Aluminium Content (Mass Fraction) Percent (Mass Fraction)	a ml	b ml
(1)	(2)	(3)	(4)
i)	Smaller than 0.05	20	0
ii)	Between 3.7 and 6.0	19	10
iii)	Between 8.0 and 11.0	18	20
iv)	Between 25 and 28.0	15	50

5.2.4 Then add 0.00 ml, 2.00 ml, 5.00 ml, 7.00 ml, 10.0 ml, 12.0 ml, 14.0 ml and 16.0 ml aliquots of the standard magnesium solution A (*see* **4.2.8**). These aliquots correspond to contents (mass fractions) in the test portion of 0.00 percent, 0.01 percent, 0.025 percent, 0.035 percent, 0.05 percent, 0.06 percent, 0.07 percent and 0.08 percent of magnesium.

For the analysis of samples having lower magnesium content, prepare two additional calibration solutions corresponding to magnesium contents (mass fractions) of 0.002 percent and 0.005 percent, by taking respectively volumes of 4.00 ml and 10.00 ml of the standard magnesium solution (*see* **4.2.9**).

5.2.5 Add 5 ml of the lanthanum solution (*see* **4.2.4**) to each flask. Dilute to the mark with water and mix.

5.3 Analysis

Measure the absorbance of the calibration solutions and the test solution(s) and taking alternate readings ensures that the settings of the burner and of the apparatus do not change during the readings (the wavelength of the line used shall be 285.21 nm).

To comply with the concentration ranges recommended by the manufacturer of the apparatus, the same dilutions for the calibration solutions and the test solution(s) shall be made if necessary.

To obtain better reproducibility and greater sensitivity, it is recommended that a slightly reducing flame be used.

5.4 Calculation

Establish a calibration graph by plotting the measured absorption of the calibration solutions against their respective contents (mass fractions).

Determine, from the measured absorbance of the test solution, the associated amount of magnesium from the calibration graph. If a number of determinations are carried out then the mean of all results shall be calculated.

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Amendments Issued Since Publication

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